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N-Ethyl-2,2-dimethyl-N-(3-methylphenyl)propanamide

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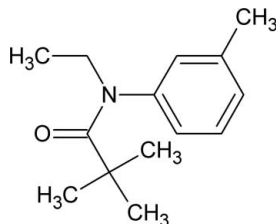
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.057; wR factor = 0.160; data-to-parameter ratio = 11.7.

In the title compound, $\text{C}_{14}\text{H}_{21}\text{NO}$, the conformation across the $\text{N}-\text{C}(\text{O})$ bond is *syn*-periplanar, the $\text{C}-\text{N}-\text{C}-\text{C}$ torsion being $-5.9(5)^\circ$. The atoms of the ethyl group attached to the N atom are disordered over two sets of sites with occupancy ratios of 0.65 (2):0.35 (2) (CH_2) and 0.689 (14):0.311 (14) (CH_3) are linked by very weak $\text{C}-\text{H}\cdots\text{O}$ interactions forming $C(8)$ chains along $[001]$. $\text{C}-\text{H}\cdots\pi$ interactions link the molecules along the *c*-axis direction.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the biological activity of amides, see: Manojkumar *et al.* (2013*a,b*). Amide groups can provide structural rigidity to molecules, see: Sreenivasa *et al.* (2013).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{21}\text{NO}$
 $M_r = 219.32$
Monoclinic, $P2_1$
 $a = 7.631(4)$ Å
 $b = 10.878(7)$ Å
 $c = 8.350(3)$ Å

$\beta = 105.60(2)^\circ$
 $V = 667.6(6)$ Å³
 $Z = 2$
Cu $K\alpha$ radiation

$\mu = 0.52$ mm⁻¹
 $T = 294$ K
 $0.22 \times 0.20 \times 0.16$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.893$, $T_{\max} = 0.921$

3786 measured reflections
2016 independent reflections
1883 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.160$
 $S = 1.06$
2016 reflections
172 parameters

55 restraints
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.34$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg is the centroid of the benzene ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C1}-\text{H1}\cdots\text{O}^i$	0.93	2.62	3.481 (2)	153
$\text{C14}-\text{H14A}\cdots\text{Cg}^{ii}$	0.96	2.85	3.769 (8)	161

Symmetry codes: (i) $x+1, y, z+1$; (ii) $x, y, z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5371).

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supporting information

Acta Cryst. (2014). E70, o223 [doi:10.1107/S1600536814001718]

N-Ethyl-2,2-dimethyl-N-(3-methylphenyl)propanamide

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S1. Comment

Amides are very common in nature, formed easily and provides structural rigidity to the molecules (Sreenivasa *et al.* 2013). Amides show a broad spectrum of pharmacological properties, including antibacterial (Manojkumar *et al.* 2013a), anti-inflammatory, antioxidant, analgesic and antiviral activity (Manojkumar *et al.* 2013b). Keeping this in mind, the crystal structure of the title compound was determined.

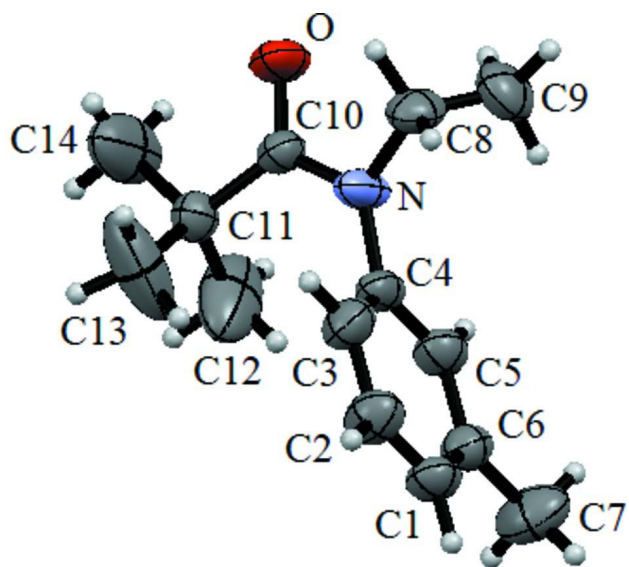
S2. Experimental

N-Ethyl-3-methylaniline (1.00g, 7.4 mmol) was taken in dry dichloromethane (10 mL) and the solution was cooled to 0 °C. To this reaction mixture 2,2-dimethylpropanoyl chloride (0.888 g, 7.4 mmol) in dichloromethane and triethylamine (1.49g, 1.48 mmol) were added slowly and the mixture was heated to 50°C for 4 hours. Reaction was monitored by TLC. Reaction mixture was cooled and washed with 10% NaHCO₃ solution. The organic layer was separated, dried and concentrated to obtained crude product which was purified by column chromatography using petroleum ether: ethyl acetate (7:3) as eluent. Yellow prisms of the title compound were obtained from slow evaporation of the solution of the compound in petroleum ether: ethyl acetate (7:3).

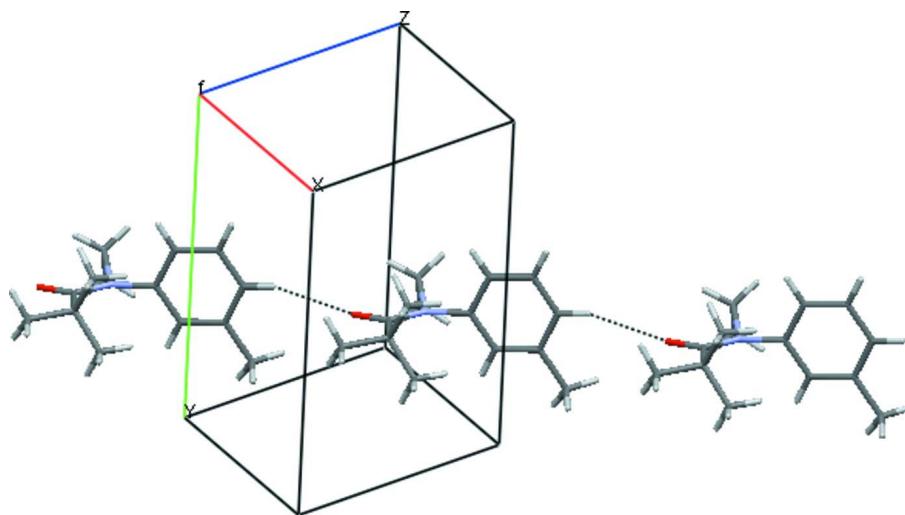
S3. Refinement

The H atoms were positioned with idealized geometry using a riding model with C-H = 0.93-0.96 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2-1.5 times of the U_{eq} of the parent atom). Flack parameter value (Flack, 1983) of 0.5 (5) was obtained in the final structure factor calculation, the presence of pseudosymmetry can lead to uncertainties about the correct space group, especially in the presence of twinning.

The C8 and C9 atoms of the ethyl group attached to N atom are disordered with site occupation factors of 0.65 (2):0.35 (2) and 0.689 (14):0.311 (14) respectively.

**Figure 1**

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. Only the major components of the disordered atoms are shown.

**Figure 2**

Molecular packing forming C(8) chains with hydrogen bonding shown as dashed lines.

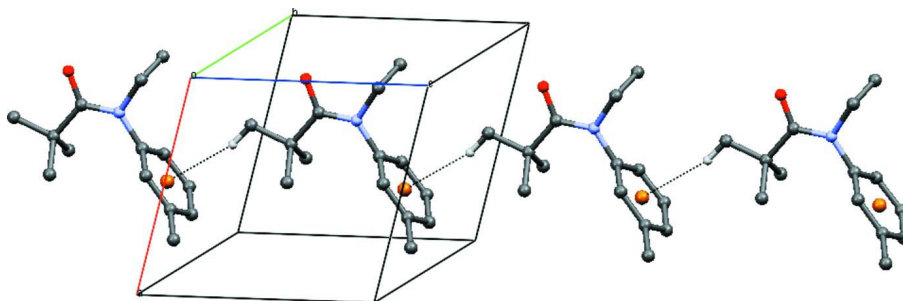


Figure 3

Stacking of molecules along c axis through C—H...Cg interactions. Cg is the centroid of the benzene ring. H-atoms not involved in H-bonding are omitted for clarity.

N-Ethyl-2,2-dimethyl-*N*-(3-methylphenyl)propanamide

Crystal data

$C_{14}H_{21}NO$

$M_r = 219.32$

Monoclinic, $P2_1$

Hall symbol: $P\ 2_1yb$

$a = 7.631\ (4)\ \text{\AA}$

$b = 10.878\ (7)\ \text{\AA}$

$c = 8.350\ (3)\ \text{\AA}$

$\beta = 105.60\ (2)^\circ$

$V = 667.6\ (6)\ \text{\AA}^3$

$Z = 2$

$F(000) = 240$

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ϕ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.893$, $T_{\max} = 0.921$

3786 measured reflections

Prism

$D_x = 1.091\ \text{Mg m}^{-3}$

Melting point: 492 K

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 172 reflections

$\theta = 5.5\text{--}65.5^\circ$

$\mu = 0.52\ \text{mm}^{-1}$

$T = 294\ \text{K}$

Prism, yellow

$0.22 \times 0.20 \times 0.16\ \text{mm}$

2016 independent reflections

1883 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 65.5^\circ$, $\theta_{\min} = 5.5^\circ$

$h = -8 \rightarrow 8$

$k = -11 \rightarrow 12$

$l = -9 \rightarrow 9$

1012 standard reflections every 2 reflections

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.160$

$S = 1.06$

2016 reflections

172 parameters

55 restraints

0 constraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.093P)^2 + 0.1495P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.34\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.16\ \text{e \AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.018 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O	0.2284 (2)	0.7636 (3)	0.6964 (2)	0.0725 (7)	
N	0.4145 (3)	0.7530 (3)	0.9461 (2)	0.0670 (8)	
C11	0.5373 (3)	0.7418 (3)	0.6942 (3)	0.0489 (6)	
C6	0.8300 (4)	0.8185 (3)	1.2843 (3)	0.0494 (6)	
C10	0.3855 (3)	0.7522 (3)	0.7806 (3)	0.0452 (6)	
C4	0.5820 (3)	0.7342 (3)	1.0730 (3)	0.0487 (7)	
C5	0.6765 (4)	0.8340 (3)	1.1519 (3)	0.0520 (7)	
H5	0.6374	0.9128	1.1164	0.062*	
C3	0.6371 (4)	0.6191 (3)	1.1298 (3)	0.0586 (8)	
H3	0.5716	0.5510	1.0789	0.070*	
C2	0.7875 (4)	0.6028 (3)	1.2607 (4)	0.0602 (8)	
H2	0.8249	0.5239	1.2975	0.072*	
C1	0.8829 (4)	0.7029 (3)	1.3376 (3)	0.0520 (7)	
H1	0.9848	0.6916	1.4271	0.062*	
C7	0.9325 (5)	0.9283 (4)	1.3690 (5)	0.0834 (11)	
H7A	0.9820	0.9109	1.4851	0.125*	
H7B	0.8515	0.9974	1.3563	0.125*	
H7C	1.0295	0.9472	1.3200	0.125*	
C14	0.4518 (6)	0.7552 (8)	0.5111 (5)	0.129 (2)	
H14A	0.5454	0.7608	0.4542	0.193*	
H14B	0.3787	0.8283	0.4908	0.193*	
H14C	0.3766	0.6849	0.4710	0.193*	
C13	0.6307 (11)	0.6209 (5)	0.7262 (9)	0.136 (3)	
H13A	0.5417	0.5564	0.7079	0.205*	
H13B	0.7044	0.6178	0.8392	0.205*	
H13C	0.7066	0.6103	0.6523	0.205*	
C12	0.6785 (8)	0.8393 (6)	0.7487 (7)	0.135 (3)	
H12A	0.7324	0.8325	0.8663	0.202*	
H12B	0.6230	0.9187	0.7237	0.202*	
H12C	0.7708	0.8293	0.6910	0.202*	
C8A	0.2598 (9)	0.7879 (9)	1.0135 (7)	0.052 (2)	0.65 (2)
H8A1	0.1744	0.8395	0.9348	0.063*	0.65 (2)
H8A2	0.3035	0.8329	1.1169	0.063*	0.65 (2)
C8B	0.2459 (15)	0.7104 (19)	1.0046 (14)	0.059 (4)	0.35 (2)
H8B1	0.1529	0.6742	0.9142	0.070*	0.35 (2)

H8B2	0.2801	0.6522	1.0958	0.070*	0.35 (2)
C9A	0.1691 (10)	0.6700 (7)	1.0428 (8)	0.087 (2)	0.689 (14)
H9A1	0.1289	0.6256	0.9400	0.131*	0.689 (14)
H9A2	0.0664	0.6887	1.0843	0.131*	0.689 (14)
H9A3	0.2542	0.6207	1.1226	0.131*	0.689 (14)
C9B	0.1877 (19)	0.8243 (14)	1.0567 (15)	0.071 (4)	0.311 (14)
H9B1	0.2883	0.8636	1.1337	0.107*	0.311 (14)
H9B2	0.0925	0.8096	1.1096	0.107*	0.311 (14)
H9B3	0.1429	0.8765	0.9617	0.107*	0.311 (14)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0395 (10)	0.130 (2)	0.0405 (9)	0.0096 (12)	−0.0029 (8)	−0.0003 (11)
N	0.0294 (10)	0.136 (2)	0.0338 (10)	0.0065 (14)	0.0055 (8)	0.0032 (14)
C11	0.0481 (13)	0.0608 (15)	0.0394 (12)	0.0028 (13)	0.0147 (10)	0.0013 (11)
C6	0.0429 (14)	0.0613 (17)	0.0417 (12)	−0.0053 (13)	0.0072 (10)	0.0004 (12)
C10	0.0375 (12)	0.0603 (14)	0.0343 (11)	−0.0002 (12)	0.0038 (9)	−0.0004 (11)
C4	0.0336 (12)	0.0806 (19)	0.0298 (10)	0.0034 (13)	0.0051 (9)	0.0036 (12)
C5	0.0456 (15)	0.0645 (17)	0.0427 (13)	0.0073 (13)	0.0063 (11)	0.0077 (12)
C3	0.0559 (17)	0.0689 (19)	0.0469 (15)	−0.0075 (15)	0.0067 (13)	−0.0085 (13)
C2	0.0631 (18)	0.0565 (17)	0.0542 (15)	0.0061 (14)	0.0040 (14)	0.0069 (13)
C1	0.0417 (15)	0.0677 (18)	0.0404 (12)	0.0050 (13)	0.0003 (11)	0.0069 (12)
C7	0.079 (2)	0.073 (2)	0.083 (3)	−0.0182 (19)	−0.0049 (19)	−0.0069 (18)
C14	0.086 (3)	0.254 (7)	0.0529 (18)	0.027 (4)	0.0323 (19)	0.021 (3)
C13	0.189 (6)	0.118 (4)	0.150 (5)	0.083 (4)	0.126 (5)	0.052 (3)
C12	0.134 (4)	0.178 (5)	0.125 (4)	−0.087 (4)	0.091 (4)	−0.062 (4)
C8A	0.038 (3)	0.075 (5)	0.045 (2)	0.006 (3)	0.0115 (19)	−0.008 (3)
C8B	0.037 (5)	0.083 (10)	0.054 (5)	−0.014 (6)	0.009 (4)	0.001 (5)
C9A	0.066 (4)	0.110 (5)	0.101 (4)	−0.013 (3)	0.049 (3)	0.001 (4)
C9B	0.057 (7)	0.095 (8)	0.073 (7)	0.006 (6)	0.035 (5)	0.003 (6)

Geometric parameters (\AA , $^\circ$)

O—C10	1.222 (3)	C7—H7C	0.9600
N—C10	1.339 (3)	C14—H14A	0.9600
N—C4	1.439 (3)	C14—H14B	0.9600
N—C8A	1.488 (7)	C14—H14C	0.9600
N—C8B	1.564 (12)	C13—H13A	0.9600
C11—C13	1.486 (5)	C13—H13B	0.9600
C11—C12	1.493 (5)	C13—H13C	0.9600
C11—C14	1.499 (5)	C12—H12A	0.9600
C11—C10	1.525 (3)	C12—H12B	0.9600
C6—C1	1.359 (4)	C12—H12C	0.9600
C6—C5	1.388 (4)	C8A—C9A	1.508 (13)
C6—C7	1.497 (5)	C8A—H8A1	0.9700
C4—C3	1.364 (4)	C8A—H8A2	0.9700
C4—C5	1.370 (4)	C8B—C9B	1.42 (3)

C5—H5	0.9300	C8B—H8B1	0.9700
C3—C2	1.367 (4)	C8B—H8B2	0.9700
C3—H3	0.9300	C9A—H9A1	0.9600
C2—C1	1.370 (4)	C9A—H9A2	0.9600
C2—H2	0.9300	C9A—H9A3	0.9600
C1—H1	0.9300	C9B—H9B1	0.9600
C7—H7A	0.9600	C9B—H9B2	0.9600
C7—H7B	0.9600	C9B—H9B3	0.9600
C10—N—C4	128.83 (19)	H7B—C7—H7C	109.5
C10—N—C8A	117.7 (3)	C11—C14—H14A	109.5
C4—N—C8A	113.3 (2)	C11—C14—H14B	109.5
C10—N—C8B	113.5 (5)	H14A—C14—H14B	109.5
C4—N—C8B	111.7 (4)	C11—C14—H14C	109.5
C13—C11—C12	107.6 (5)	H14A—C14—H14C	109.5
C13—C11—C14	109.0 (4)	H14B—C14—H14C	109.5
C12—C11—C14	108.8 (4)	C11—C13—H13A	109.5
C13—C11—C10	111.7 (3)	C11—C13—H13B	109.5
C12—C11—C10	112.4 (3)	H13A—C13—H13B	109.5
C14—C11—C10	107.3 (2)	C11—C13—H13C	109.5
C1—C6—C5	119.1 (3)	H13A—C13—H13C	109.5
C1—C6—C7	120.8 (3)	H13B—C13—H13C	109.5
C5—C6—C7	120.1 (3)	C11—C12—H12A	109.5
O—C10—N	117.2 (2)	C11—C12—H12B	109.5
O—C10—C11	119.2 (2)	H12A—C12—H12B	109.5
N—C10—C11	123.6 (2)	C11—C12—H12C	109.5
C3—C4—C5	119.1 (2)	H12A—C12—H12C	109.5
C3—C4—N	121.1 (3)	H12B—C12—H12C	109.5
C5—C4—N	119.3 (3)	N—C8A—C9A	106.8 (6)
C4—C5—C6	120.6 (3)	N—C8A—H8A1	110.4
C4—C5—H5	119.7	C9A—C8A—H8A1	110.4
C6—C5—H5	119.7	N—C8A—H8A2	110.4
C4—C3—C2	120.7 (3)	C9A—C8A—H8A2	110.4
C4—C3—H3	119.6	H8A1—C8A—H8A2	108.6
C2—C3—H3	119.6	C9B—C8B—N	100.9 (13)
C3—C2—C1	119.9 (3)	C9B—C8B—H8B1	111.6
C3—C2—H2	120.1	N—C8B—H8B1	111.6
C1—C2—H2	120.1	C9B—C8B—H8B2	111.6
C6—C1—C2	120.6 (2)	N—C8B—H8B2	111.6
C6—C1—H1	119.7	H8B1—C8B—H8B2	109.4
C2—C1—H1	119.7	C8A—C9A—H9A1	109.5
C6—C7—H7A	109.5	C8A—C9A—H9A2	109.5
C6—C7—H7B	109.5	H9A1—C9A—H9A2	109.5
H7A—C7—H7B	109.5	C8A—C9A—H9A3	109.5
C6—C7—H7C	109.5	H9A1—C9A—H9A3	109.5
H7A—C7—H7C	109.5	H9A2—C9A—H9A3	109.5
C4—N—C10—O	175.9 (3)	C8B—N—C4—C5	−109.8 (9)

C8A—N—C10—O	−9.8 (6)	C3—C4—C5—C6	2.2 (4)
C8B—N—C10—O	25.7 (9)	N—C4—C5—C6	174.5 (2)
C4—N—C10—C11	−5.9 (5)	C1—C6—C5—C4	−1.7 (4)
C8A—N—C10—C11	168.3 (5)	C7—C6—C5—C4	179.8 (3)
C8B—N—C10—C11	−156.1 (9)	C5—C4—C3—C2	−1.8 (4)
C13—C11—C10—O	−116.5 (5)	N—C4—C3—C2	−174.0 (3)
C12—C11—C10—O	122.5 (4)	C4—C3—C2—C1	0.9 (5)
C14—C11—C10—O	2.9 (5)	C5—C6—C1—C2	0.8 (4)
C13—C11—C10—N	65.3 (5)	C7—C6—C1—C2	179.3 (3)
C12—C11—C10—N	−55.7 (5)	C3—C2—C1—C6	−0.4 (4)
C14—C11—C10—N	−175.2 (4)	C10—N—C8A—C9A	94.8 (5)
C10—N—C4—C3	−88.2 (4)	C4—N—C8A—C9A	−90.0 (5)
C8A—N—C4—C3	97.3 (5)	C8B—N—C8A—C9A	4.1 (8)
C8B—N—C4—C3	62.4 (9)	C10—N—C8B—C9B	−107.2 (7)
C10—N—C4—C5	99.6 (4)	C4—N—C8B—C9B	97.4 (8)
C8A—N—C4—C5	−74.9 (5)	C8A—N—C8B—C9B	−2.2 (6)

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the benzene ring.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C1—H1 \cdots O ⁱ	0.93	2.62	3.481 (2)	153
C14—H14A \cdots Cg ⁱⁱ	0.96	2.85	3.769 (8)	161

Symmetry codes: (i) *x*+1, *y*, *z*+1; (ii) *x*, *y*, *z*+1.